

Ultra-high temperature emittance measurements for space and missile applications

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Advanced modeling and design efforts for many aerospace components require high temperature emittance data. Applications requiring emittance data include propulsion systems, radiators, aeroshells, heatshields/thermal protection systems, and leading edge surfaces. The objective of this work is to provide emittance data at ultra-high temperatures. MSFC has a new instrument for the measurement of emittance at ultra-high temperatures, the Ultra-High Temperature Emissometer System (Ultra-HITEMS). AZ Technology Inc. developed the instrument, designed to provide emittance measurements over the temperature range 700-3500K. The Ultra-HITEMS instrument measures the emittance of samples, heated by lasers, in vacuum, using a blackbody source and a Fourier Transform Spectrometer. Detectors in a Nicolet 6700 FT-IR spectrometer measure emittance over the spectral range of 0.4-25 μm . Emitted energy from the specimen and output from a Mikron M390S blackbody source at the same temperature with matched collection geometry are measured. Integrating emittance over the spectral range yields the total emittance. The ratio provides a direct measure of total hemispherical emittance. Samples are heated using lasers. Optical pyrometry provides temperature data. Optical filters prevent interference from the heating lasers. Data for Inconel 718 show excellent agreement with results from literature and ASTM 835. Measurements taken from levitated spherical specimens provide total hemispherical emittance data; measurements taken from flat specimens mounted in the chamber provide near-normal emittance data. Data from selected characterization studies will be presented. The Ultra-HITEMS technique could advance space and missile technologies by advancing the knowledge base and the technology readiness level for ultra-high temperature materials.

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Emittance

- *Directional v. Hemispherical*
 - *Directional illumination and hemispherical collection or vice versa*
 - *Near-normal is a special case of directional where the angle of incidence is near the normal vector of the sample surface*
 - *Hemispherical illumination and hemispherical collection*
 - *Just about impossible to build a reflectance mode instrument that does this*
 - *Classically determined by calorimetry*
- *Flat surface (ESL) → near-normal emittance*
- *Sphere (ESL) → hemispherical emittance*

Other options for high temperature measurements

- *Other High Temperature Emissometers*

	<i>Temp range</i>	<i>Mode</i>	<i>Spectral range</i>	<i>Angular Extent</i>
NPL	150 to 1800 °C	direct	0.6 to 9.6-um	0 to 70°
Advanced Fuel Research	100 to 1000 (2000) °C	both	0.8 to 20-um	na
Southern Research Institute	1500 to 5000 °F	direct	not applicable	na
BNM-LNE	ambient – TBD	reflected	0.8 to 14-um	12, 24, 36, 48, 60°

- *Alternative strategies*
 - Calorimetry
 - Pyrometry
 - Advantages and disadvantages

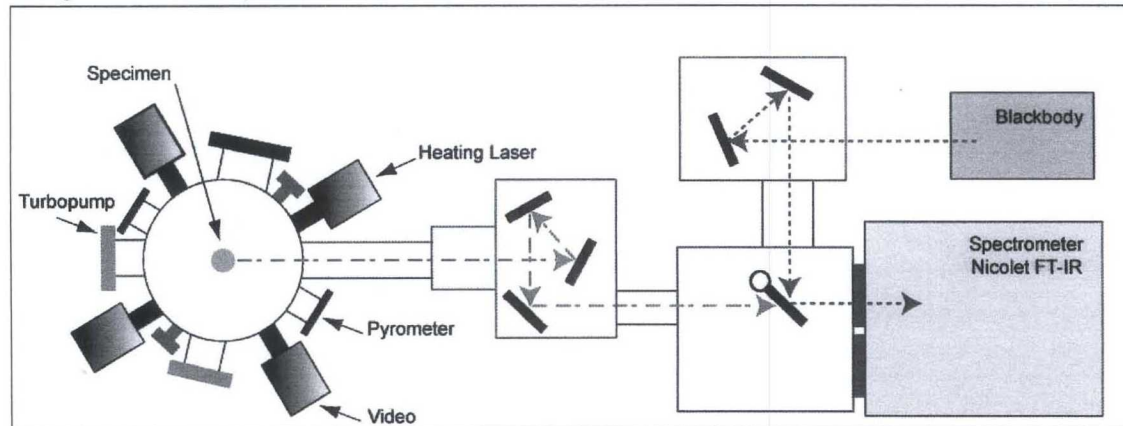
Ultra-High Temperature Emissivity Measurement System (Ultra HITEMS) Background

- Emissivity data at operating temperature needed for thermal design.
- System originally developed for measurement of small ($\sim 2\text{mm}$), smooth levitated specimens.
- New customers (J-2X and CEV) have requirements for larger, flat samples with special surface treatments.
- The instrument is being adapted to meet the new requirements.
- New apparatus, optics and protocols are being developed/implemented to meet new customer needs.



Ultra-High Temperature Emissivity System

Configuration A: Blackbody Calibration



Configuration B: Emissivity Measurements

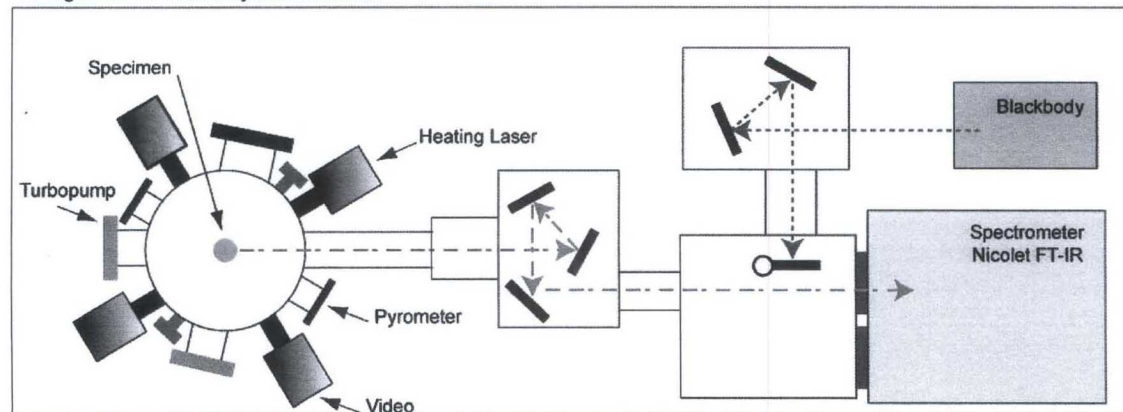
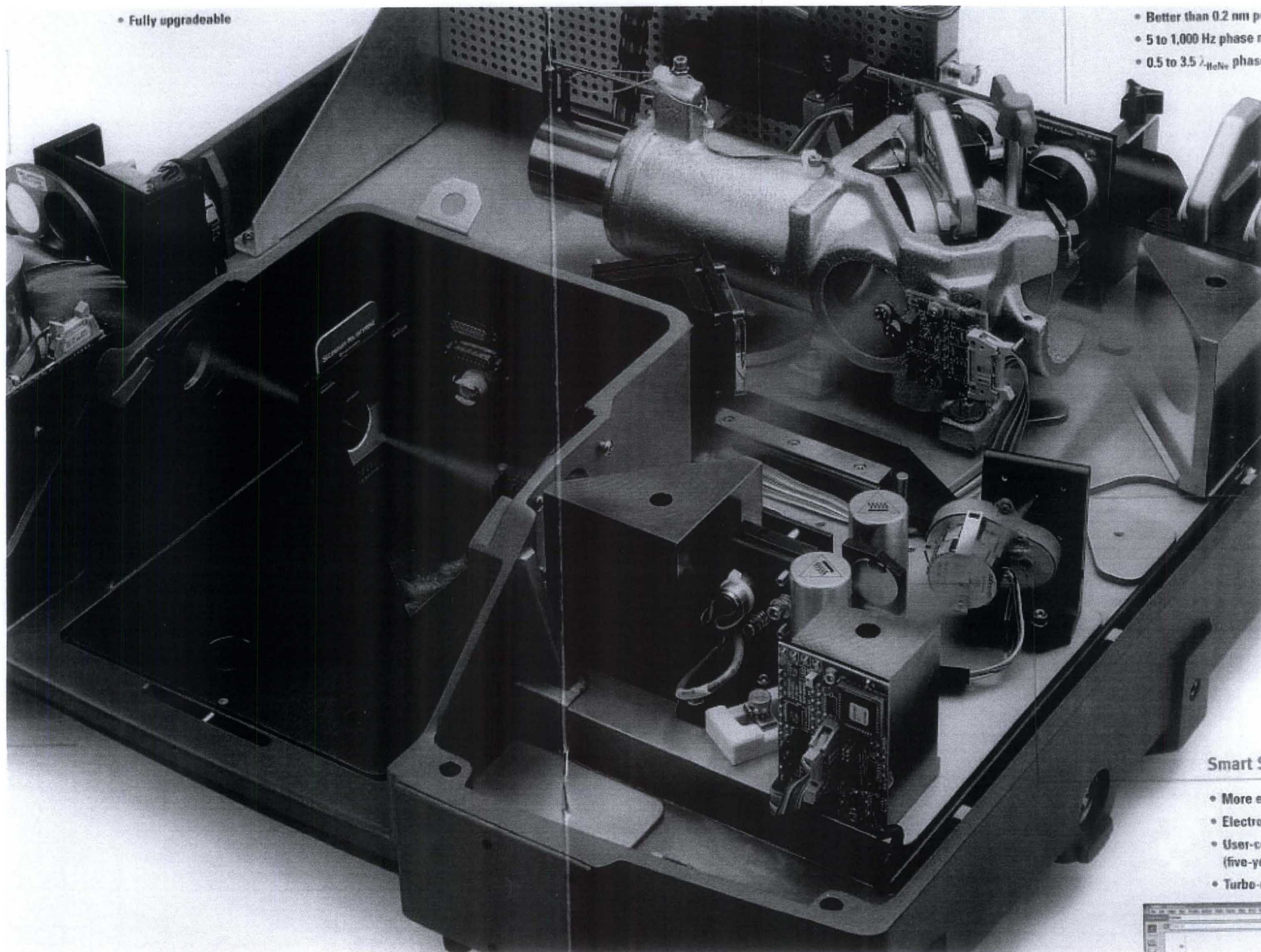


Figure 1. Schematic of the Emissivity Measurements

• Fully upgradeable

• Better than 0.2 nm pc
• 5 to 1,000 Hz phase m
• 0.5 to 3.5 λ_{HeNe} phase



Smart S

• More ef
• Electron
• User-co
(five-ye
• Turbo-n

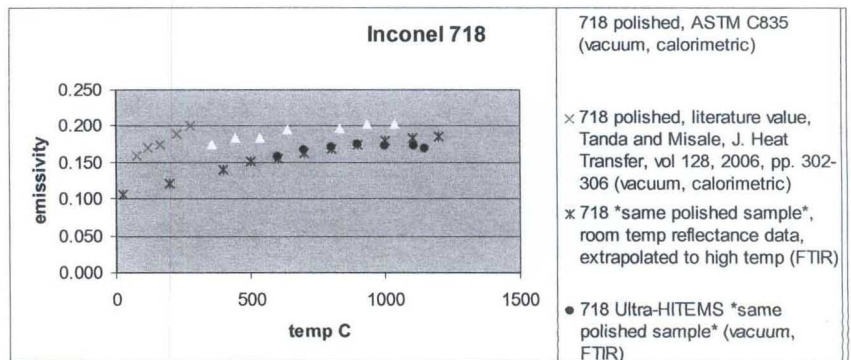
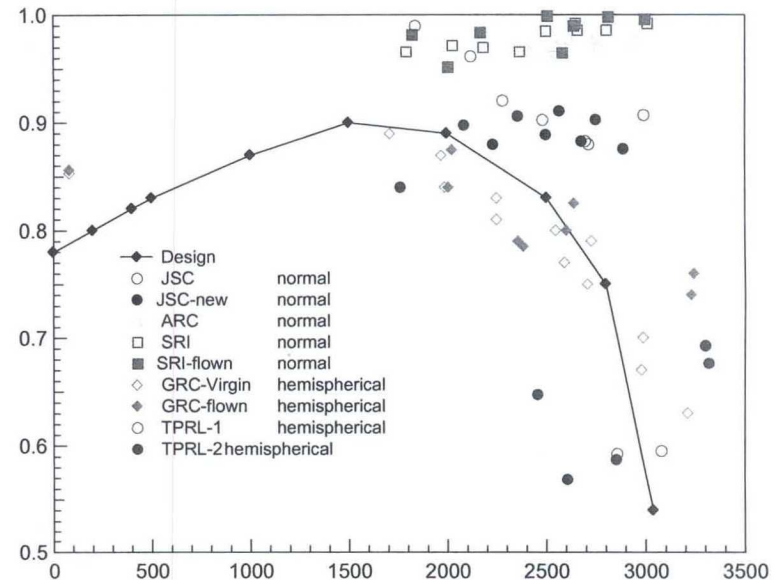


Designed to minimize uncertainty

- *Commonality between reference/background paths and sample paths*
 - Same collection geometries
 - Matched radiance
 - *Same solid angle collection*
 - *Same area collection*
 - Same number of transport altering surfaces
 - Goal to maximize common path
 - Equivalent path lengths
 - Purge benefit
 - Effect of violating these on uncertainty
 - Sensitivity
 - Calibration factors

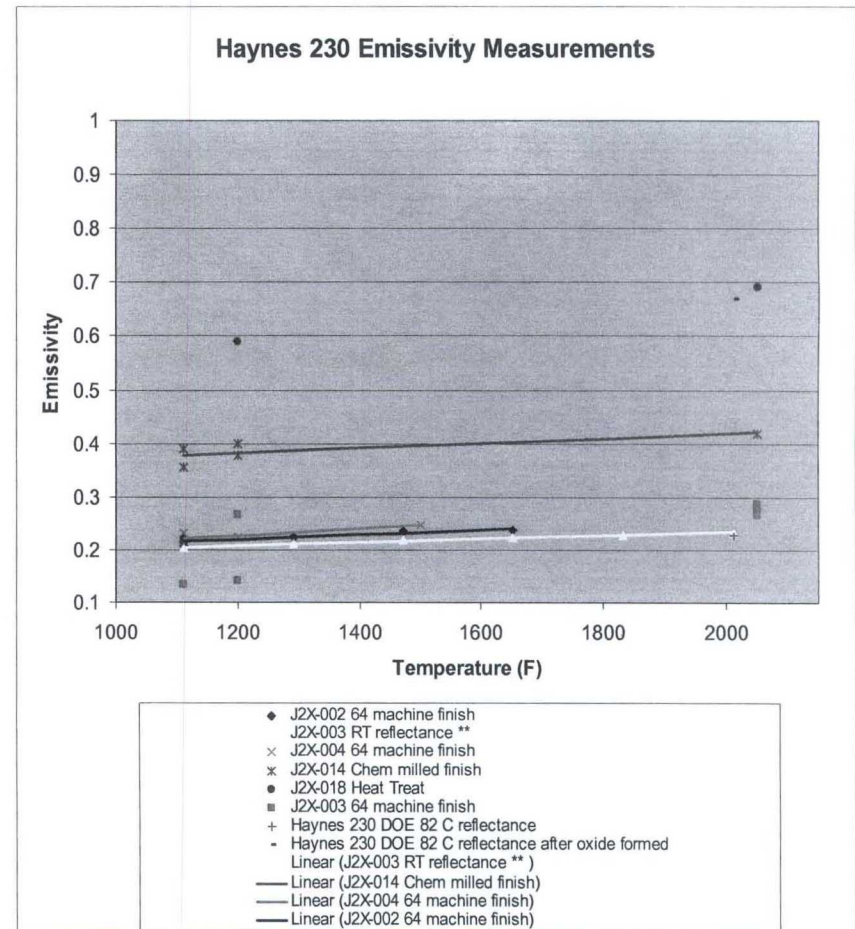
HITEMS Calibration

- No clear calibration standards for high temperature emissivity. Very difficult to achieve quantitative comparisons between different labs, using different test conditions and different samples. An example of data for different samples of, measured in different labs RCC is shown in the adjacent graph.
- Polished Inconel 718 used for initial calibration. Results shown in graph below
 - Ni-based alloy with published emissivity data for polished surface available
 - Polished surface chosen for reproducibility
- High emissivity calibration material is being sought. Materials examined include SiC and graphite. An AZ tech coating was also evaluated, but it was not stable in test conditions.
- Hexoloy SA SiC from St. Gobain has been used as a high emissivity calibration material.
 - Additional emissivity tests are planned using this calibration factor

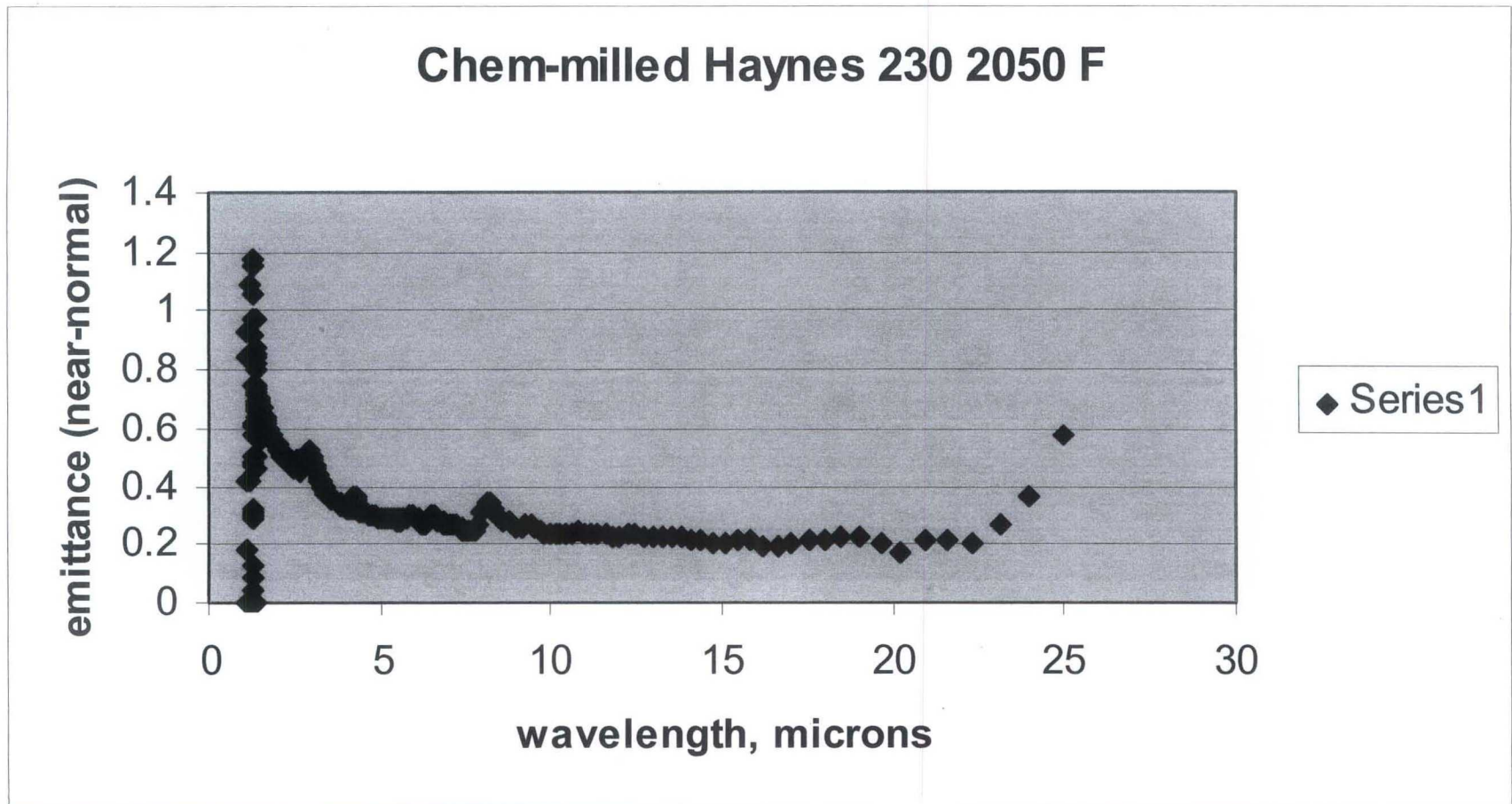


Emissivity Measurements Haynes 230 HITEMS, and reflectance data from AZ Tech and DOE report

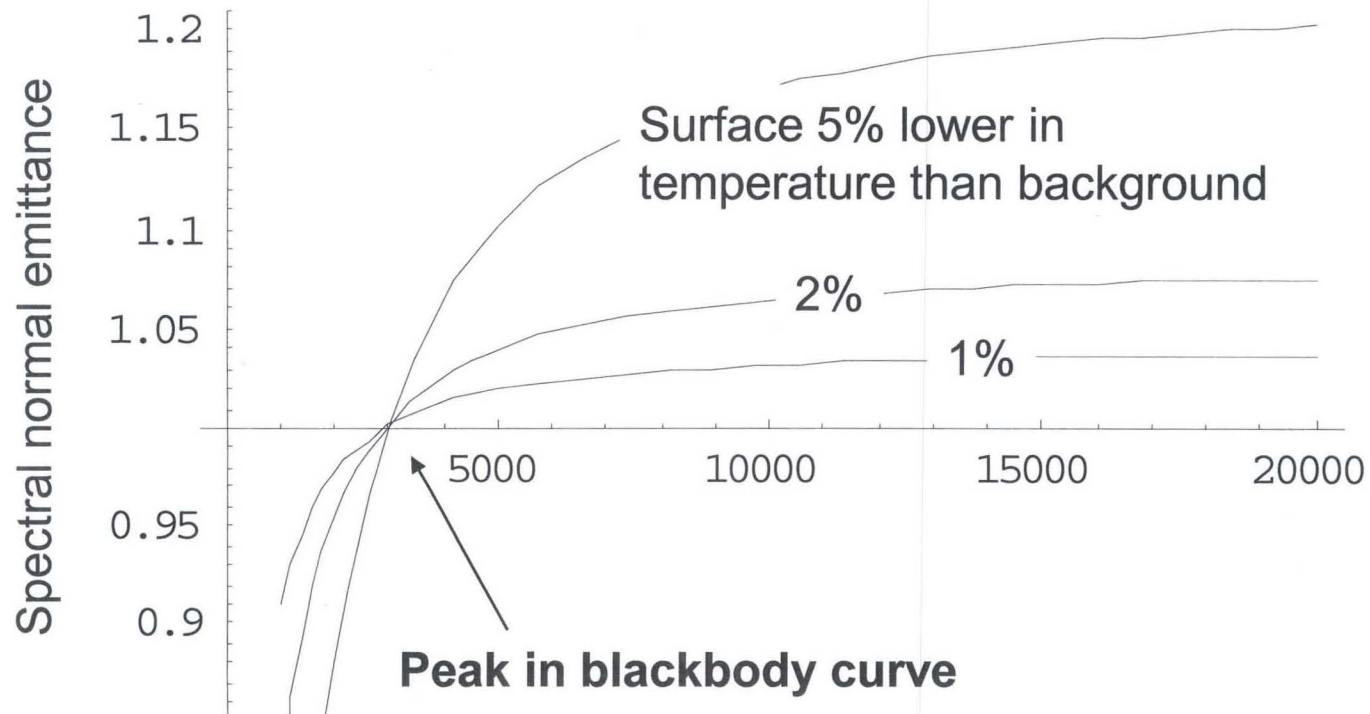
- HITEMS from heating at 2050 F and $\sim 10^{-6}$ Torr data for Haynes 230 is in good agreement with data from room temperature reflectance data from AZ Tech on same samples
- HITEMS data for Haynes 230 appears to be in good agreement with reflectance-based data Taken at 82 C from DOE study with 500 hour vacuum anneal at 2012 F and $\sim 10^{-5}$ Torr.
- The 0.23 value from the DOE study based on measurements prior to the anneal
- The 0.67 value from the DOE study is from the same sample, after the anneal. The authors of the study report the formation of an oxide layer on the surface of the sample during the anneal which changed the emissivity.
- This value is similar to the value from the HITEMS for a sample which had been heat treated in hydrogen at 2000 F and in air at 1950 F. The sample had a visible oxide layer.



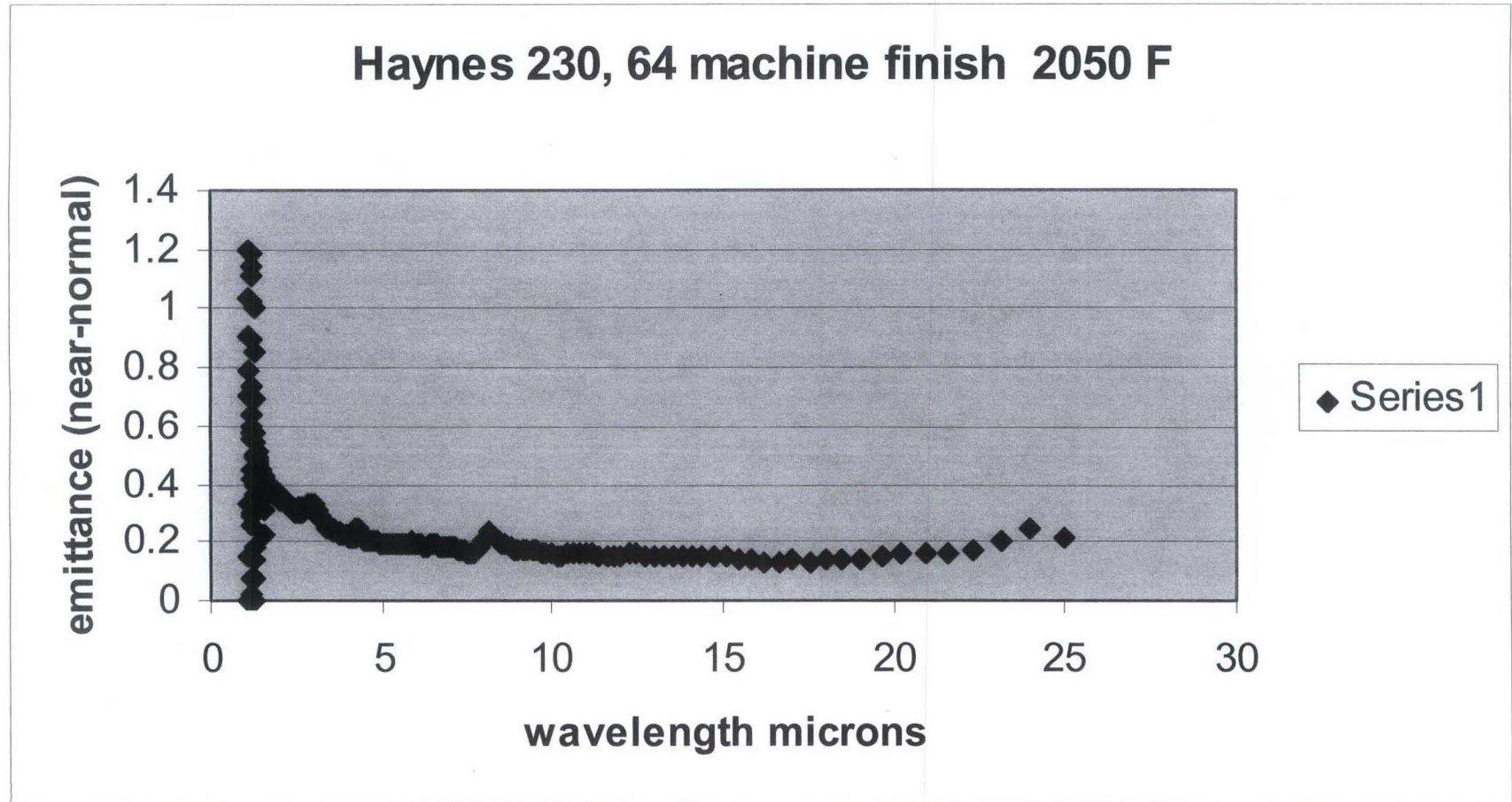
Near-normal spectral emittance for Haynes 230, Chem-milled



Error due to Temperature Mismatch



Near-normal spectral emittance for Haynes 230, 64 machine finish



Future Plans

- Continue efforts to improve, understand and validate the HITEMS system
 - This test method can provide the required spectral data in relevant environment
 - Use of extrapolated reflectance data has been suggested, but issues can arise when materials and materials properties change at high temperature. For reactive systems, this is very likely.
 - Post-test reflectance data could identify changes in the material but would not be able to predict emissivity at temperature.
- Lack of accepted calibration standard(s) for material
 - Significant discrepancies exist in among data sets from different labs
 - Some options proposed by NIST and are being explored